# Chapter 9
# Final Waste Form Requirements of Low-Level Radioactive Waste

## 9-1. 10 CFR 61 Requirements

*a. Waste characteristic requirements.* Waste form requirements that must be met in order for radioactive waste to be disposed of in shallow land burial are detailed in 10 CFR 61.56. This part of the regulation is quoted in full below. The following requirements are minimum requirements for all classes of waste and are intended to facilitate handling at the disposal site and provide protection of health and safety of personnel at the disposal site.

(1) Waste must not be packaged for disposal in cardboard or fiberboard boxes.

(2) Liquid waste must be solidified or packaged in sufficient absorbent material to absorb twice the volume of the liquid.

(3) Solid waste containing liquid shall contain as little free-standing and noncorrosive liquid as is reasonably achievable, but in no case shall the liquid exceed 1 percent of the volume.

(4) Waste must not be readily capable of detonation or of explosive decomposition or reaction at normal pressures and temperatures, or of explosive reaction with water.

(5) Waste must not contain, or be capable of generating, quantities of toxic gases, vapors, or fumes harmful to persons transporting, handling, or disposing of the waste. This does not apply to radioactive gaseous waste packaged in accordance with paragraph *9-1.a(7)* of this section.

(6) Waste must not be pyrophoric. Pyrophoric materials contained in waste shall be treated, prepared, and packaged to be nonflammable.

(7) Waste in a gaseous form must be packaged at a pressure that does not exceed 1.5 atmospheres at 20 °C. Total activity must not exceed 100 curies per container.

(8) Waste containing hazardous, biological, pathogenic, or infectious material must be treated to reduce to the maximum extent practicable the potential hazard from the nonradiological materials.

*b. Stability requirements.* The requirements in this section are intended to provide stability of the waste. Stability is intended to ensure that the waste does not structurally degrade and affect the overall stability of the site through slumping, collapse, or other failure of the disposal unit and thereby lead to water infiltration. Stability is also a factor in limiting exposure to an inadvertent intruder, since it provides a recognizable and nondispersible waste.

(1) Waste must have structural stability. A structurally stable waste form will generally maintain its physical dimensions and its form, under the expected disposal conditions such as weight of overburden and compaction equipment, the presence of moisture, and microbial activity, and internal factors such as radiation effects and chemical changes. Structural stability can be provided by the waste form itself, processing the waste to a stable form, or placing the waste in a disposal container or structure that provides stability after disposal.

(2) Notwithstanding the provisions in 61.56(a)(2) and (3), liquid wastes, or wastes containing liquid, must be converted into a form that contains as little free-standing and noncorrosive liquid as is reasonably achievable, but in no case shall the liquid exceed 1 percent of the volume of the waste when the waste is in a disposal container designed to ensure stability, or 0.5 percent of the volume of the waste for waste processed to a stable form.

(3) Void spaces within the waste and between the waste and its package must be reduced to the extent practicable.

## 9-2. NRC Requirements

*a. Introduction.* The NRC has issued a technical position paper on waste form requirements that was initially developed in 1983 to provide guidance to both fuel-cycle and non-fuel-cycle waste generators on waste form test methods and results acceptable to the NRC staff for implementing the 10 CFR 61 waste form requirements. This position paper has an appendix which provides special requirements for cement solidified waste. The following material is taken from the “Technical Position on Waste Form: Revision I" (NRC 1991).

*b. Class A waste stability requirements.* Solidified Class A waste products which are segregated from Class B and C wastes (Classes A, B, and C of LLRW are defined by 10 CFR 61 and EM 11 10-35-1) should be free-standing monoliths and have no more than

0.5 percent of the waste volume as free liquids as measured using the method described in American Nuclear Society (ANS) 55.1 (ANS 1979). Class A waste products which are not segregated from Class B and C wastes should meet the stability guidance for Class B and C wastes as provided below.

*c. Class B and C waste stability requirements.* The stability requirements for Class B and C solidified wastes put forth by the NRC deal with compressive strength, thermal degradation, radiation degradation, biodegradation resistance, leach testing, immersion testing, pH range, full-scale testing, and homogeneity as described below.

(1) Compressive strength. Solidified waste specimens should have compressive strengths of at least 60 psi when tested in accordance with ASTM C39 (ASTM 1979b). Compressive strength tests for bituminous products should be performed in accordance with ASTM D 1074 (ASTM 1980). Many solidification agents (such as cement) will be easily capable of meeting the 60-psi limit for properly solidified wastes. For such cases, process control parameters should be developed to achieve maximum practical compressive strengths, not simply to achieve the minimum acceptable compressive strength. Please refer to the NRC requirements for cement-solidified wastes in Section 9-2.d.

(2) Thermal degradation. Waste specimens should be resistant to thermal degradation. The heating and cooling chambers used for thermal degradation testing should conform to the description given in ASTM B553 (ASTM 1979a). Samples suitable for performing compressive strength tests in accordance with ASTM C39 (ASTM 1979b) or ASTM D1074 (ASTM 1980) should be used. Samples should be placed in the test chamber and a series of 30 thermal cycles carried out in accordance with ASTM B553. The high temperature limit should be 60 °C, and the low temperature limit should be -40 °C. Following testing, the waste specimens should have the maximum practical compressive strengths.

(3) Radiation degradation. The specimens for each proposed waste stream formulation should remain stable after being exposed to a radiation field equivalent to the maximum level of exposure expected from the proposed wastes to be solidified. Specimens for each proposed waste stream formulation should be exposed to a minimum of $10^8$ rads in a gamma irradiator or equivalent. If the maximum level of exposure is expected to exceed $10^8$ rads, testing should be performed at the expected maximum accumulated dose. Following irradiation, the irradiated specimens should have the maximum practical compressive strengths.

(4) Biodegradation resistance. Specimens for each proposed waste stream formulation should be tested for resistance to biodegradation in accordance with both ASTM G21 (ASTM 1970) and ASTM G22 (ASTM 1976). No indication of culture growth should be visible. Specimens should be suitable for compression testing in accordance with ASTM C39 or ASTM D 1074, as applicable. Following the biodegradation testing, specimens should have the maximum practical compressive strengths. For polymeric or bitumen products, some visible culture growth from contamination, additives, or biodegradable components on the specimen surface that does not relate to overall substrate integrity may be present. For these cases, additional testing should be performed. If culture growth is observed upon completion of the biodegradation test for polymeric or bitumen products, the test specimens should be removed from the culture and washed free of all culture and growth with water, with only light scrubbing. An organic solvent compatible with the substrate may be used to extract surface contaminants. The specimen should be air-dried at room temperature and the test repeated. Specimens should have observed culture growths rated no greater than 1 in the repeated ASTM G21 test. The specimens should have no observed growth in the repeated ASTM G22 test. Compression testing should be performed in accordance with ASTM C39 and ASTM D1074, as applicable, following the repeated G21 and G22 tests. The minimum acceptable compressive strength for bituminized waste forms is 60 psi. Compressive strengths should be established for other media. If growth is observed following the extraction procedure, longer term testing of at least 6 months should be performed to determine biodegradation rates. The Bartha-Pramer method (Bartha and Pramer 1965) is acceptable for this testing. Soils used should be representative of those at disposal facilities. Biodegradation extrapolated for full-size waste forms to 300 years should produce less than a 10-percent loss of the total carbon in the waste form.

(5) Leach testing. Leach testing should be performed for a minimum of 90 days in accordance with the procedure in ANS 16.1 (ANS 1981). Specimen sizes should be consistent with the samples prepared for the ASTM C39 or ASTM D 1074 compressive strength tests. In addition to the demineralized water test specified in the ANS 16.1, additional testing using other leachants specified in the Standard should also be performed to confirm the solidification agent's leach resistance in other leachant

media. It is preferred that the synthesized seawater leachant also be tested. In addition, it is preferable that radioactive tracers be utilized in performing the leach tests. For proposed nuclear power station waste streams, cobalt, cesium, and strontium should be used as tracers. The leachability index, as calculated in accordance with ANS 16.1, should be greater than 6.0.

(6) Immersion testing. Waste specimens should maintain maximum practical compressive strengths as tested using ASTM C39 or ASTM D 1074, following immersion for a minimum period of 90 days. Immersion testing may be performed in conjunction with leach testing.

(7) pH range. Waste specimens should have less than O.5 percent by volume of the waste specimen as free liquids, as measured using the method described in ANS 55.1 (ANS 1979). Free liquids should have a pH between 4 and 11. (For cement-solidified water, free liquids should have a minimum pH of 9.)

(8) Full-scale testing. If small, simulated laboratory size specimens are used for the above testing, test data from sections or cores of the anticipated full-scale products should be obtained to correlate the characteristics of actual size products with those of simulated laboratory size specimens. This testing may be performed on nonradioactive specimens. Correlation testing should be performed using 90-day immersion (including post-immersion compression) tests on the most conservative waste stream(s) intended for use for the particular solidification medium; i.e., the waste stream that presents the most difficulty in consistently producing a stable product. The full-scale specimens should be fabricated using solidification equipment the same as or comparable to that used for processing actual LLRW in the field.

(9) Homogeneity. Waste samples from full-scale specimens should be destructively analyzed to ensure that the product produced is homogeneous to the extent that all regions in the product can expect to have compressive strengths representative of the compressive strength as determined by testing lab-scale specimens. Full-scale specimens may be fabricated using simulated nonradioactive products; however, the specimens should be fabricated using solidification equipment that is the same as or comparable to that used in the field for actual LLRW.

*d. Cement stabilization requirements.*

(1) Introduction.

(a) Portland and pozzolonic cements have been observed to exhibit unique chemical and physical interactive behavior when used with certain materials and chemicals encountered in some low-level radioactive waste streams. Therefore, cement waste form qualifications will be specifically addressed. This discussion is not intended to be applied generically to all stabilization agents and is intended to provide information on an acceptable approach for demonstrating that a cement-solidified low-level radioactive waste form will possess the long-term (300-year) structural stability that is required by 10 CFR 61 for Class B and Class C wastes.

(b) Low-level radioactive waste generators/processors may perform qualification testing, as described below, to qualify recipes for a range of waste compositions (concentrations and loadings) for a given type of waste stream. It is incumbent upon the party providing 10 CFR 20.311 certification, however, to show that the composition of the waste form specimens used in the qualification testing adequately covers the range of waste compositions that will be encountered in the field. An acceptable approach to qualification testing is to perform the tests not only at the maximum waste loading but also at lower loadings (at least one), with appropriate variations in water/cement ratios and proportions of additives. It should not be necessary to perform all the qualification tests for all of the waste loadings, but adequate justifications should be provided for any omissions. Each individual waste stream should be qualified with test data obtained for that specific waste stream. In cases where two or more waste streams are combined, it should be demonstrated that the specimen compositions used in the qualification testing adequately cover the range of compositions that are intended to be stabilized in the field. This may be accomplished by performing the full series of qualification tests on the "worst-case" composition only, along with one or more tests on alternate compositions, sufficient to show that the selected "worst-case" was chosen correctly.

(2) Qualification test specimen preparation.

(a) The method used to prepare the test specimens is extremely important because the test specimens will predict the performance of the full-size waste products. Experience has shown that the method employed in mixing the ingredients can have a dramatic influence on the reactivity of the materials, the structure of the solidified waste form, and the resultant properties and characteristics of the waste form. Important parameters include the type of equipment and mixing time because they will determine the amount of energy imparted to the ingredients used in the solidification recipe. In preparing the laboratory specimens, it should be shown by analysis and/or testing that the type of mixing equipment used, the mixing time, the speed of the mixer, etc., will, in combination, impart the same degree of mixing to the lab specimens as to the full-size product. It should also be shown that the degree of mixing is sufficient to ensure production of homogeneous waste forms.

(b) The curing conditions for small, lab-scale specimens should, to the extent practical, be the same as the conditions obtained with full-scale products. Because of the exothermic heat of the hydration reaction in cement waste forms, the interior temperature of a full-size waste form significantly elevates. To mimic this condition, it is recommended that the specimens be cured in a suitable oven for a period of time equivalent to the time required for the center-line temperature of a full-scale waste form to decrease to a near-ambient (30 ‘C or lower) temperature level.

(c) The compressive strength of hydrated cement and concrete solids increases asymptotically as the mixtures cure. Normally, the strength at 28 days approaches 75 percent or more of the peak value; however, when pozzolonic cements are used, the time required to reach peak strength may be extended. Sufficient test specimens should be prepared to determine the compressive strength increase with time to ensure that the specimens have attained greater than 75 percent of the projected peak strength prior to subjecting the remaining specimens to qualification testing.

(3) Compressive strength.

(a) For solidification agents that are easily capable of meeting the 60-psi minimum compressive strength, the waste forms should achieve maximum practical compressive strengths. Portland cement mortars are readily capable of achieving compressive strengths of 5,000 to 6,000 psi, which is approximately two orders of magnitude greater than the minimum compressive strength required to resist deformation under load in current low-level waste burial trenches. Thus, a mean compressive strength equal to or greater than 500 psi is recommended for waste form specimens cured for a minimum of 28 days.

(b) Compressive strengths of cement-stabilized waste forms should be determined in accordance with procedures described in ASTM C39 (ASTM 1979b). It is recommended that the compressive strength test specimens be right circular cylinders, 5 to 7 cm (2 to 3 in. ) in diameter, with a length-to-diameter ratio of approximately 2. Because hydrated cement solids are brittle ceramic materials that fail in tension or shear rather than compression, and at regions of localized stress concentration or microstructural flaw, there tends to be considerable scatter in the strength test data, even if all processing variables are kept relatively constant. Therefore, sufficient specimens should be tested to determine the mean compressive strength and standard deviation. Because of the many variables involved, a decision regarding the specific number of specimens to be tested is left to the judgement of the waste processor/qualifier. In no case, however, should the number of as-cured compressive strength test specimens be less than ten. Compressive strength tests should be performed after the qualification test specimens have been allowed to cure for approximately 24 hr.

(4) Thermal degradation.

(a) It is important for cement-stabilized LLRW forms to be resistant to thermal degradation in order to retain structural stability. The thermal cycling test imposes a stress among the various microconstituents of the waste form and between different regions of the waste form. By cycling between the maximum and minimum temperatures called for in the test, any cracks initiated in the test specimen may propagate and eventually measurably weaken the waste form. The extent of any degradation that might occur will be a function of various factors such as the amount of cementitious material in the waste form, the bond strength between the materials present, and the morphology of the microconstituents in the waste form microstructure. Thus, the thermal cycling test challenges the structural capability of the specimens and serves as a very useful vehicle for screening out unfavorable “weak” formulations.

(b) The heating and cooling chambers used in determining the thermal cycling resistance of cement-stabilized waste forms should conform to the description given in ASTM standard B553 (ASTM 1979a). However, because that test method addresses thermal cycling of electroplated plastics, not cement-solidified waste materials, some modifications to the test procedure are necessary. Test specimens suitable for performing compressive strength tests in accordance with ASTM C39 should be used. The specimens should be tested while not in a container. A series of 30 thermal cycles should be carried out with the provision that the specimens should be allowed to come to thermal equilibrium at the high (60 °C) and low (-40 °C) temperature limits.

Thermal equilibrium should be confirmed by measurements of the center temperature of at least one specimen. A minimum of three specimens for each waste formulation should be subjected to the thermal cycling tests. Following the thermal cycling tests, the specimens should be visually examined and should be free of any evidence of significant cracking, spalling, or bulk disintegration. If there are no significant visible defects, the test specimens should be subjected to compression strength testing in accordance with ASTM C39 and should have mean compressive strengths that are equal to or greater than 500 psi.

(5) Radiation degradation.

(a) Since cementitious materials are not affected by gamma radiation to greater than $10^9$ rads, which is considerably in excess of the test requirement of $10^8$ rads, irradiation testing need not be conducted on cement-stabilized waste forms unless the following conditions exist:

The waste form contains ion exchange resins or other organic media.

The expected cumulative dose on waste forms containing other materials is greater than $10^9$ rads.

Testing should be performed on specimens exposed to the following:

$10^8$ rads or the expected maximum dose greater than $10^8$ rads for waste forms that contain ion exchange resins or other organic media.

An expected maximum dose greater than $10^9$ rads for other waste forms.

In cases where irradiation testing is warranted, a minimum of three specimens should be tested for each waste formulation being qualified.

(b) Following the irradiation exposure, the specimens should be examined visually and should be free of any evidence of significant cracking, spalling, or bulk disintegration. If there are no significant visible defects, the test specimens should be subjected to compressive strength testing in accordance with ASTM C39 and should have mean compressive strengths that are equal to or greater than 500 psi (3.45 mPa).

(6) Biodegradation.

(a) As indicated in 10 CFR 61, a structurally stable waste form is one that will be relatively unaffected by microbial activity. Experience in biodegradation testing of cement-stabilized waste forms has shown that they generally do not support fungal or bacterial growth. The principal reason for this appears to be that the fungi and microbes used in the G21 and G22 (ASTM 1970, 1976) tests require a source of carbon for growth. In the absence of any carbonaceous materials in the waste stream, there is no internal food source available for culture growth. Consequently, biodegradation qualification testing for cement-stabilized waste forms need not be conducted unless the waste form contains carbonaceous material.

(b) For cement-stabilized waste forms containing carbonaceous materials, there should be no evidence of culture growth during the G21 and G22 tests. The test specimens should also be free of any evidence of significant cracking, spaling, or bulk disintegration. At least three specimens should be tested for each organic waste stream formulation. If there are no significant visible effects following the test exposures, the test specimens should be subjected to compression strength testing in accordance with ASTM C39 and should be shown to have mean compressive strengths equal to or greater than 500 psi.

(7) Leach testing.

(a) The leach testing procedure is test ANS 16.1 (ANS 1981). A test specimen is completely immersed in a measured volume of water which is changed on a prescribed schedule. Upon removal, the leachant is analyzed for the radionuclides of interest. As prescribed in the standard, a leachability index is calculated and should be greater than 6.0. The leachant specified in ANS 16.1 is deionized water. Additional testing using other leachants should also be performed to confirm the solidification agent's leach resistance in other leachant media. Synthesized seawater leachant is listed as a preferred leachant alternative. For reasons of economy, it is desirable to limit the bulk of the testing to one leachant. If it can be shown that the chosen leachant is the most aggressive one, testing with one leachant is appropriate. Sufficient preliminary testing should be conducted to identify the most aggressive leachant for each waste form formulation being qualified. An acceptable method for identifying the most aggressive leachant is to perform 24 hr or longer leaching measurements on both leachants and to use the leachant that resulted in the lowest leach indices for the remaining days of testing.

(b) The period of time specified for the leach test is a minimum of 90 days. This time period was selected as a means of determining whether there might be a change in leach mechanism with time; early leach rates are most often explained by diffusion, while other mechanisms such as erosion, dissolution, or corrosion would generally be discernible only after longer leaching times. However, any leaching that involves mechanisms such as erosion, dissolution, corrosion, or other physical or chemical phenomena would most likely be readily observed visually and through mechanical testing. Such observations would be made as part of the immersion test.

(8) Immersion testing.

(a) No standard method of immersion testing has been adopted for LLRW. Immersion testing may be performed in conduction with leach testing. Immersion testing should be performed for a minimum period of 90 days. Immersion testing should be performed in either deionized water or synthesized seawater. The immersion liquid should be selected on the basis of 24-hr or longer leach tests that identify the most aggressive immersion medium.

(b) At least three specimens should be used for each formulation being qualified. Test specimens should be cured for a minimum of 28 days prior to being immersed. Following immersion, the specimens should be examined visually and should be free of any evidence of significant cracking, spalling, or bulk disintegration. If there are no significant visible effects, the specimens should be subjected to compression strength testing in accordance with ASTM C39 and should have post-immersion mean compressive strengths that are equal to or greater than 500 psi and not less than 75 percent of the pre-immersion mean compressive strength. If the post-immersion mean compressive strength is less than 75 percent of the as-cured specimens' pre-immersion mean compressive strength, but not less than 500 psi, the immersion testing interval should be extended to a minimum of 180 days. Additional specimens should be used. For these cases, sufficient compressive strength testing should be conducted (for example, after 120, 150, and 180 days of immersion) to establish that the compressive strengths level off and do not continue to decline with time.

(c) Certain waste streams such as bead resins, chelates, filter sludges, and floor drain wastes have been found to exhibit complex relationships of cure time and immersion resistance. For these streams, additional immersion testing should be performed on specimens that have been cured in sealed containers for a minimum of 180 days. The immersion period should be for a minimum of 7 days followed by a drying period of 7 days in ambient air at a minimum temperature of 20 'C. After the specimens are dried, they should meet the post-immersion test visual and compressive strength criteria specified above.

(9) pH range. Waste test specimens should have less than 0.5 percent by volume of the waste volume as free liquids as measured using the method described in ANS 55.1 (ANS 1979). As cement is an alkaline material, evidence of acidic free liquids is indicative of improper waste form preparation or curing. Therefore, any free liquid from cement-stabilized waste forms should have a minimum pH of 9.

(10) Full-scale testing. It is necessary to correlate the characteristics of full-size products with those of laboratory size specimens. The correlation of full-scale product characteristics should be accomplished by performing compressive strength tests on material cured for a minimum of 28 days and 90-day immersion tests that include post-immersion compressive strength tests for the most conservative waste stream being qualified.